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FORM PTO-1390 (Modified) (REV 5-93) U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371

ATTORNEY'S DOCKET NUMBER
017447/0170

U.S. APPLICATION NO. (If known, see 37 C.F.R. 1.5)
09/720730

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/JP99/03407	June 25, 1999	June 29, 1998

TITLE OF INVENTION
SPUTTERING TARGET

APPLICANT(S) FOR DO/EO/US
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Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a FIRST submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
   - ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
   - ☒ has been transmitted by the International Bureau.
   - ☐ is not required, as the application was filed in the United States Receiving Office (RO/US)
6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
   - ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
   - ☐ have been transmitted by the International Bureau.
   - ☐ have not been made; however, the time limit for making such amendments has NOT expired.
   - ☒ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☒ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern other document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☒ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A FIRST preliminary amendment.
    ☐ A SECOND or SUBSEQUENT preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☐ Other items or information

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U.S. APPLICATION NO. (If known, see 37 C.F.R. 1.50)	INTERNATIONAL APPLICATION NO.	ATTORNEY'S DOCKET NUMBER
Unassigned 09/720730	PCT/JP99/03407	017447/0170

17. ☒ The following fees are submitted:	CALCULATIONS	PTO USE ONLY
Basic National Fee (37 CFR 1.492(a)(1)-(5): Search Report has been prepared by the EPO or JPO $860.00		
International preliminary examination fee paid to USPTO (37 CFR 1.482) $690.00		
No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2) $710.00		
Neither international preliminary examination fee (37 CFR 1.482) nor International search fee (37 CFR 1.445(a)(2)) paid to USPTO $1,000.00		
International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4) $100.00		
ENTER APPROPRIATE BASIC FEE AMOUNT =	$860.00	
Surcharge of $130.00 for furnishing the oath or declaration later than 20 Months from the earliest claimed priority date (37 CFR 1.492(e))		

Claims	Number Filed		Included in Basic Fee		Extra Claims		Rate		
Total Claims	21	-	20	=	1	×	$18.0	$18.00	
Independent Claims	3	-	3	=	0	×	$80.0	$0.00	
Multiple dependent claim(s) (if applicable)							$270.0		

TOTAL OF ABOVE CALCULATIONS =	$878.00	
Reduction by ½ for filing by small entity, if applicable. Verified Small Entity statement must also be filed. (Note 37 CFR 1.9, 1.27, 1.28).	$0.00	
SUBTOTAL =	$878.00	
Processing fee of $130.00 for furnishing English translation later the 20 months from the earliest claimed priority date (37 CFR 1.492(f). +		
TOTAL NATIONAL FEE =	$878.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). $40.00 per property +	$40.00	
TOTAL FEES ENCLOSED =	$918.00	

				Amount to be: refunded	$
				charged	$

a. ☒ A check in the amount of $918.00 to cover the above fees is enclosed.

b. ☐ Please charge my Deposit Account No. 19-0741 in the amount of $954.00 to the above fees. A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 19-0741. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

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REGISTRATION NUMBER 25,479

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Atty. Dkt. No. 017447/0170

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Koichi WATANABE et al.

Title: SPUTTERING TARGET

Appl. No.: Unassigned

Filing Date: 12/29/2000

Examiner: Unassigned

Art Unit: Unassigned

PRELIMINARY AMENDMENT

Commissioner for Patents
Washington, D.C. 20231

Sir:

Prior to examination of the above-identified application, Applicants respectfully request that the following amendments be entered into the application:

IN THE CLAIMS:

Please delete claims 2 and 19 without prejudice.

REMARKS

Entry of the foregoing amendments prior to examination is respectfully requested.

Respectfully submitted,

Date December 29, 2000

By Johnny A. Kumar 34,649

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1/PRTS

SPUTTERING TARGET

Technical Field

The present invention relates to a Nb sputtering target suitable for forming liner material of a semiconductor element or the like.

Background Art

In recent years, semiconductor industry typical in LSIs is increasing rapidly. In semiconductor elements of 64Mbit DRAMs and subsequent ones, the higher the integration, reliability and functionality become, the higher accuracy is demanded for microfabrication technology. With such higher densification of an integrated circuit, a width of metal interconnection formed essentially of Al or Cu is becoming 1/4 μm or less.

On the other hand, to operate an integrated circuit at a high speed, it is indispensable to reduce resistance of an Al interconnection or a Cu interconnection. In the existing interconnection structure, the interconnection resistance is generally reduced due to an increase in the height of the interconnection. However, in semiconductor devices where further higher integration and densification are achieved, an existing stacking structure causes a poor coverage of an insulating film formed on the interconnection, resulting naturally in a deterioration of yield. Accordingly, the interconnection technology of the devices itself is demanded to improve.

Accordingly, dual damascene (DD) interconnection technology different from the existing one is under study. In the DD interconnection technology, first, on a substrate film thereon an interconnection trench is previously formed, metal essentially consisting of Al or Cu that is interconnection material is deposited by use of sputtering method or CVD method to form a film. Then, after letting fill the interconnection metal in the trench due to heat treatment (reflow), an excess of the interconnection metal is removed by use of CMP (Chemical Mechanical Polishing) method or the like.

Here, in a DD interconnection structure, how to excellently fill Al or the like in the interconnection trench is of importance. As filling technology, the aforementioned reflow technology or the like can be applied. As a film improving reflow characteristic of Al (liner film), a Ti film is generally used. However, the Ti film, in the step of reflow, reacts with Al to form Al_3Ti compound to result in a remarkable increase of the interconnection resistance.

In view of these, the liner materials to Al in the place of Ti are variously under study. Among these, Nb in particular is reported to be effective in use. Nb, in comparison with Ti, can reduce the interconnection resistance and improve reflow characteristics of Al.

When considering an application in the next generation semiconductor memories such as DRAMs of an integration rate of 256Mbit or 1Gbit or more, resistivity of an interconnection film is demanded to be $4\mu\Omega$cm or less, for

instance. However, when with a Nb film deposited by use of an existing Nb target as the liner material, thereon an Al film or Al alloy film is deposited to form an Al interconnection film, it is difficult to suppress the resistivity to $4 \mu \Omega$cm or less with reproducibility.

In addition, though being necessary to suppress admixture of dust in the interconnection film as much as possible, in the existing Nb target, there is a problem that giant dust such as exceeding 1 μm for instance occurs suddenly. As a result, product yield of the semiconductor devices remarkably deteriorates.

As mentioned above, in the Al interconnection film having the Nb liner film formed by use of the existing Nb target, the resistivity of for instance $4 \mu \Omega$cm or less that is demanded for the DRAMs of 256Mbit or 1Gbit or more can not be satisfied sufficiently and with reproducibility. Further, during deposition of the Nb liner film, the giant dust suddenly occurs to result in a decrease of the product yield of the semiconductor devices. Thus, the existing Nb target faces a difficulty in applying in the next generation semiconductor memories or the like.

An object of the present invention is to provide a sputtering target that, in applying the DD interconnection technology to form an Al interconnection film, enables to heighten electrical characteristics and quality of the Nb film as liner material of the Al film. In specific, the object is to provide a sputtering target that enables to obtain with reproducibility a Nb film capable of suppressing

the resistivity of an Al interconnection film to for instance $4\mu\Omega$cm or less. In addition, another object is to provide a sputtering target enabling to suppress a sudden occurrence of the giant dust and to improve the product yield of Nb films.

Disclosure of the Invention

A first sputtering target of the present invention is characterized in consisting essentially of high purity Nb of which Ta content is 3000ppm or less, wherein dispersion of the Ta contents over the whole target is within ±30%.

A second sputtering target of the present invention is characterized in consisting essentially of high purity Nb, wherein each grain of the Nb has a grain diameter in the range of 0.1 to 10 times an average grain diameter and ratios of grain diameters of adjacent grains are in the range of 0.1 to 10. The second sputtering target is further characterized in that dispersion of the ratios of grain diameters of adjacent grains over the whole target is within ±30%.

A third sputtering target of the present invention is characterized in consisting essentially of high purity Nb of which oxygen content is 200ppm or less, wherein dispersion of the oxygen contents over the whole target is within ±80%.

Ta contained in the Nb target is easily oxidized than Nb is and oxides of Ta (such as Ta_2O_5 or the like) are very stable. Furthermore, above a certain temperature, Ta reacts easily with Al to form an intermetallic compound such as

Al_3Ta. Such oxides and intermetallic compounds in the interconnection cause an increase of the resistivity.

In the first sputtering target, a content of Ta as an impurity is set at 3000ppm or less. In addition, the dispersion of Ta content over the whole target is set within ±30%. According to the high purity Nb target in which the Ta content is reduced, a Ta content in a Nb film deposited therewith can be suppressed low. Accordingly, an increase of the resistivity of an interconnection film due to generation of Ta_2O_5 or Al_3Ta can be largely suppressed.

In the third sputtering target, a content of oxygen as an impurity is set at 200ppm or less. Further, the dispersion of the oxygen content over the whole target is set within ±80%. A reduction of the oxygen content in the high purity Nb target enables to suppress a content of oxygen in a Nb film deposited therewith. Thereby, Ta_2O_5 causing an increase of the resistivity in the interconnection film can be suppressed from generating, resulting in realization of low resistivity of the interconnection film.

Reasons incapable of suppressing with reproducibility the resistivity of the interconnection film deposited by use of the existing Nb target to for instance $4\mu\Omega$cm or less are due to the aforementioned Ta contained in Nb. The present inventors first found this fact. The sputtering target of the present invention is achieved by finding an influence of Ta in particular Ta oxides such as Ta_2O_5 existing in a Nb film on the interconnection film.

As to the giant dust (particles) generated suddenly

from the existing Nb target, it was found that when grains try to recover strain due to a thermal influence, difference in amounts of the strain due to size difference of grains causes the sudden generation of the giant dust. That is, when there is a large difference between grain sizes of adjacent grains, in the course of a larger grain recovering strain, smaller grains existing adjacent thereto undergo large stress. As a result, part of the smaller grains or the smaller grains themselves scatter to stick on a substrate as the giant dust.

In the second sputtering target of the present invention, the ratios of grain sizes of the adjacent grains are set in the range of 0.1 to 10. By thus reducing the ratio of grain sizes of the adjacent grains, stress difference generated in the course of the grains recovering the strain can be alleviated. Thereby, the giant dust can be suppressed from occurring to result in a remarkable increase of the yield of the Nb films or interconnection films comprising the same.

Brief Description of the Drawing

Fig. 1 is a diagram showing one example of configuration of a semiconductor device having a DD interconnection structure deposited by use of a sputtering target of the present invention.

Modes for Implementing the Invention

In the following, modes for implementing the present

invention will be explained.

A sputtering target of the present invention consists of high purity Nb. It is said that an amount of impurity element in the sputtering target is generally desirable to reduce. In the present invention, an impurity element influencing particularly on characteristics of a Nb film deposited with a Nb target of the present invention is found. Based on the above finding, an amount of a particular element is reduced and dispersion of the amount of the impurity element is suppressed low.

In concrete, a sputtering target of the present invention consists of high purity Nb of which Ta content is 3000ppm or less. Further, the sputtering target of the present invention consists desirably of high purity Nb of which oxygen content is 200ppm or less.

Since Nb and Ta are in an adjacent relationship in the periodic table, Nb raw material necessarily contains Ta. These Nb and Ta are generally called refractory metals and, being together 5A group elements, have very similar properties. Accordingly, it is not easy to separate Ta from Nb and resultantly ordinary Nb material contains relatively large amount of Ta as an impurity element.

However, Ta is easily oxidized and the resultant Ta oxide such as Ta_2O_5 is very stable. In addition, Ta reacts easily with Al to form an intermetallic compound such as Al_3Ta above a certain temperature. These, oxide and intermetallic compound containing Ta, when existing in an interconnection film, play a role of increasing the

resistivity.

When Ta is contained much in the Nb target or there is a large dispersion of the Ta content, within the Nb film deposited therewith or at an interface with Al film deposited thereon, Ta_2O_5 or Al_3Ta are rapidly formed to result in an increase of the resistivity of the interconnection film. Actually, in a trench interconnection a Nb film is deposited due to the sputtering, thereafter an Al-Cu film is formed. The interface thereof is investigated. As the result, Ta_2O_5 is detected much and such Al interconnection film is found to be high in the resistivity. From the above results, the reason of incapability of suppressing with reproducibility the resistivity of the interconnection film using the existing Nb target to for instance $4\mu\Omega$cm or less is found to come from Ta contained in the Nb, further oxygen causing Ta_2O_5 or the like to form.

Accordingly, in the present sputtering target, a Ta content in Nb constituting the target is set at 3000ppm or less. Thus, by suppressing the Ta content in the Nb target to 3000ppm or less, an amount of Ta in a Nb film deposited therewith can be reduced. Accordingly, an amount of Ta_2O_5 or Al_3Ta formed or existing within the Nb film or at an interface with an Al film deposited thereon can be largely reduced.

Furthermore, in the present sputtering target, an oxygen content in Nb constituting the target is set at 200ppm or less. The Nb film deposited with the Nb target like this is low in the oxygen content that is one reason of generating

Ta oxide (Ta_2O_5 or the like). Thereby, an amount of Ta_2O_5 precipitating at an interface between the Nb film and an Al film (or Al alloy film) deposited thereon can be remarkably reduced.

Thereby, the resistivity of the interconnection film comprising the Nb film can be largely reduced. In particular, it largely contributes in reducing the resistivity of the Al interconnection film having the Nb film as liner material. The Ta content in the sputtering target is preferable to be 2000ppm or less, and further preferable to be 1000ppm or less. The oxygen content in the sputtering target is preferable to be 150ppm or less, and further preferable to be 100ppm or less. Thereby, the resistivity of the interconnection film can be further reduced.

Dispersion of the Ta content in the present sputtering target is preferable to be within ±30% as a whole target. Thus, by suppressing the dispersion of the Ta content low over the whole target, the resistivity over the whole interconnection film deposited therewith can be lowered with reproducibility. The dispersion of the Ta content over the whole target is further preferable to be within ±15%.

Dispersion of the oxygen content in the sputtering target of the present invention is preferable to be within ±80% as the whole target. Thus, by suppressing the dispersion of the oxygen content low over the whole target, the resistivity of the whole interconnection film deposited therewith can be reduced with reproducibility. The dispersion of the oxygen content over the whole target is

further preferable to be within ±50%, and still further preferable to be within ±30%.

The aforementioned dispersions of the Ta and oxygen contents indicate values obtained in the following ways. For instance, from a surface of a sputtering target of a diameter of 320 to 330mm, specimens for analysis are sampled from 9 points. The respective specimens are sampled from a center of the target and the respective positions of ±75mm and ±150mm respectively in the directions of X and Y axes with respect to the center. Of these 9 specimens for analysis, the Ta content and oxygen content are measured. From the minimum and maximum values thereof, with the following equation, dispersion is obtained.

Dispersion [%] = {(maximum value-minimum value)/(maximum value + minimum value)} × 100

The Ta content is measured with ICP-AES (inductively coupled plasma-atomic emission spectroscopy) that is usually used. The oxygen content is measured with LECO' inert gas fusion/infrared absorption spectroscopy. Sampling positions on the target are appropriately adjusted according to the target size.

Concerning other impurities than Ta and oxygen in the sputtering target of the present invention, up to an approximate level of general-purpose high purity metal material, a slight amount can be acceptable. However, in view of lowering the interconnection resistance, other elements are also preferable to be reduced similarly.

The present sputtering target consisting of high purity

Nb is further preferable to control the size of the Nb grains constituting the target in the following way. That is, a grain diameter of each grain is in the range of 0.1 to 10 times an average grain diameter and ratios of grain diameters of adjacent grains is in the range of 0.1 to 10.

There are many reports on the relationship between the grain diameter of a target and the dust. Usually, the so-called dust is generated in the following ways. In one way, grains flied off during the sputtering adhere to an adhesion preventive plate or a non-erosion area of the target disposed in a sputtering apparatus. These peel off in flake to generate the dust. In the other way, due to a potential difference generated in a gap between the grains, there occurs an abnormal discharge to result in generation of molten grains called splash. Usually ones of which size is approximately 0.2 to 0.3 μm are called the dust.

However, the dust suddenly generated from the existing Nb sputtering target is as large as 1 μm or more in size, which is remarkably large in comparison with that from the existing one. The shape is also as massive as rock is. As the result of various investigations of the massive dust, it is found in a mode such that part of the grain or grain itself is extracted due to the sputtering. The inventors of the present invention carried out investigations while paying attention to the grain sizes of the adjacent grains and found that when there is a large difference in grain diameters of adjacent grains, the aforementioned giant dust occur.

That is, a target surface undergoes a considerable

thermal influence due to the sputtering and the respective grains try to recover the strain thereof. An amount of the strain that each grain possesses differs depending on the size thereof. In the course of the larger grain recovering the strain, smaller grains, when existing adjacent to the larger grain, are subjected to much stress. As the result, there occurs a phenomenon that part of the smaller grain or the smaller grain itself flies off.

When due to the difference of grain sizes of the adjacent grains like this the part of the grain or the grain itself flies off, it adheres on a substrate as giant dust to result in a decrease of yield of the Nb film. Accordingly, in the present sputtering target, a ratio of the grain sizes of the adjacent grains is set in the range of 0.1 to 10.

By setting the ratio of grain sizes of the adjacent grains 10 times or less or 1/10 or more, in the course of the grains undergone the thermal influence trying to recover the strain, difference of stress can be alleviated. Thereby, the part of the grain or the grain itself can be prevented from flying off. As the result, the giant dust is suppressed from occurring and the yield of the Nb film or the interconnection film comprising the Nb film can be largely improved. The grain size ratio of the adjacent grains is preferable to be in the range of 0.5 to 5, and further preferable to be in the range of 0.5 to 1.5.

The dispersion of the grain size ratios of the adjacent grains is preferable to be within ±30% the target as a whole. Thus, by suppressing the dispersion of the grain size ratios

low over the whole target, the Nb film deposited therewith as a whole can suppress the giant dust from occurring. The dispersion of the grain size ratios of the adjacent grains over the whole target is preferable to be within ±15%, and further preferable to be within ±10%.

The grain size ratio of the adjacent grains can be obtained in the following ways. First, a straight line is drawn on a grain structure micrograph taken under an arbitrary magnification (optical micrograph under a magnification of 200 times, for instance). Of 30 grains on the straight line and adjacent with each other, grain diameters (in this case, the grain diameter denotes a diameter of the minimum circle circumscribing one grain) are measured. The aforementioned grain size ratio denotes a ratio of grain sizes of adjacent grains in this case.

The dispersion of the grain size ratios denotes a value obtained in the following ways. For instance, for analysis, 9 specimens are sampled from a surface of a sputtering target of a diameter of 320 to 330mm. The specimens each are sampled from the center of the target and positions located at 75mm and 150mm from the center in directions of X-axis and Y-axis relative to the center, respectively. The grain size ratio is measured of each of these 9 specimens for analysis. The dispersion is obtained from the maximum and minimum values thereof with the following formula.

Dispersion [%] = {(the maximum value - the minimum value)/(the maximum value + the minimum value)} × 100

For the Nb grains in the sputtering target, as

mentioned above, the grain size ratios between the adjacent grains are particularly important to be in the range of 0.1 to 10. However, when the total dispersion of grain diameters of the Nb grains is large, there are many grains different in sputtering rate to result in larger steps between the adjacent grains. Accordingly, the grain diameters of the Nb grains are set in the range of 0.1 to 10 times an average grain diameter.

The concrete average grain diameter of the Nb grains is preferable to be in the range of 100 μm or less. When the average grain diameter exceeds 100 μm, the dust increases to result in a larger dispersion of the interconnection resistance of the obtained thin films. The average grain diameter of the Nb grains is preferable to 75 μm or less, and further preferable to be 50 μm or less.

The average grain diameter of the Nb grains denotes a value obtained in the following ways. First, similarly with the case of measurement of the composition dispersion, specimens are sampled from the surface of the sputtering target. Each specimen is polished, followed by etching with an etching solution of HF: HNO_3: H_2O = 2: 2: 1, thereafter observed of texture by use of an optical microscope. On a micrograph taken under a magnification of 200 times, a circle of a diameter of 50mm is drawn. The number of grains contained in the circle and not traversed by a circumference (number of grains: A) and number of grains traversed by the circumference (number of grains: B) are counted. Based on [total number of grains in the circle = the number A + number

B/2], an area per a grain is calculated. Assuming a section of one grain a circle, the average grain diameter is calculated as the diameter thereof.

The sputtering target of the present invention can be manufactured in the following ways for instance.

First, high purity Nb that is formation raw material of a sputtering target is prepared. In specific, Nb_2O_5 containing concentrate of which Ta content is 3000ppm or less is chemically processed to be high purity oxide. Then, by making use of thermite reduction method due to Al, crude metal Nb is obtained. This is for instance electron beam melted (EB) to refine high purity Nb.

Here, a step of melting due to the EB melting, with intentions of reducing the Ta content and the dispersion thereof, and further with an intention of reducing the oxygen content and the dispersion thereof, is preferable to repeat multiple times. To reduce the dispersion of the Ta content, it is also effective to uniformly disperse Ta existing in Nb due to zone refining method.

Next, to the obtained Nb ingot, plastic working due to forging and rolling is applied. The working rate during the plastic working is set at for instance 50 to 98%. According to the plastic working of the working rate like this, an adequate amount of heat energy can be given to the ingot. Due to the energy, Ta or oxygen can be homogenized (reduction of the dispersion). In the step of plastic working, as demands arise, intermediate heat treatment can be implemented.

The energy given by the aforementioned plastic working

destroys grains of the ingot. Furthermore, it works effectively in removing minute internal defects. Thereafter, at a temperature of approximately 800 to 1300°C, heat treatment is applied for 1 hour or more. By applying the heat treatment to the Nb material of which grains are once destroyed due to the plastic working to make the Nb grain structure recrystallize, the grain diameters of the Nb grains can be controlled. In specific, the grain diameter of each grain can be made to be in the range of 0.1 to 10 times an average grain diameter and the grain size ratios of the adjacent grains can be made to be in the range of 0.1 to 10. The recrystallization contributes to reduce the dispersion of the Ta content and oxygen content.

Thus obtained high purity Nb raw material is machined into a desired disc-like shape and bonded with a backing plate consisting of for instance Al. In bonding with the backing plate, the diffusion bonding due to the hot pressing is preferably applied. The temperature during the diffusion bonding is preferable to be in the range of 400 to 600°C. Because, thereby, Al of which melting point is 660°C is prevented from plastically deforming, diffusion of Ta atoms and oxygen atoms in the target is suppressed from occurring, and further an adverse effect on the grain diameter of the Nb grain in the target is suppressed from exerting. The raw material obtained here is machined into a prescribed size thereby a sputtering target of the present invention can be obtained.

The sputtering target of the present invention, though

capable of using for forming interconnection films of various kinds of electronic devices, can be particularly preferably used in forming a Nb film as liner material to an Al film (or Al alloy film). A Nb film sputter deposited with a sputtering target of the present invention is 3000ppm or less in Ta content, and further 2000ppm or less, 1000ppm or less, and the dispersion thereof is within ±30%, and further within ±15%. The oxygen content is 200ppm or less, and further 150ppm or less, 100ppm or less, and the dispersion thereof is within ±80%, and further within ±50%, within ±30%. In addition, number of the dust (giant dust in particular) is remarkably scarce.

The Nb film like this is, as mentioned above, suitable for the liner material of the Al interconnection. By giving an Al film or Al alloy film on the Nb film obtained due to the present invention, an interconnection film can be constituted. According to such interconnection film, an interconnection structure suitable for applying the DD interconnection technology can be provided. Thereby, the resistivity such as $4\mu\Omega$cm or less for instance that is demanded for DRAMs of 256Mbit or 1Gbit can be satisfied sufficiently and with reproducibility. This largely contributes in suppressing signal delay. Further, high density interconnection can be realized with high reliability and reproducibility. This largely contributes in improving the yield of the interconnection films.

The interconnection films such as mentioned above can be used in various kinds of electronic components typical in

semiconductor devices. In specific, the semiconductor devices such as ULSIs and VLSIs, and electronic components such as SAW devices, TPHs and LCD devices can be cited.

Fig. 1 is a sectional view showing one example of configuration of a semiconductor device comprising a DD interconnection structure having a Nb film deposited by use of the sputtering target of the present invention. In Fig. 1, reference numeral 1 denotes Si substrate thereon an element configuration is formed. On the Si substrate 1, an insulating film 2 is formed, thereon 2 a first Al interconnection 3 being formed connected with an element structure through a contact hole (not shown in the figure).

On the insulating film 2 having the first Al interconnection 3 an interlayer dielectric film 4 is formed, thereon 4 interconnection trenches 5 (5a, 5b and 5c) being disposed. The interconnection trenches 5a, 5b and 5c are disposed on a surface side of the interlayer dielectric film 4. The interconnection trench 5b is formed so as to reach the first Al interconnection 3, that is an interconnection hole (via hole).

In each of the interconnection trenches 5a, 5b and 5c, first a Nb film 6 deposited by use of the present sputtering target is formed as liner material. On the Nb film 6, a second Al interconnection 7 consisting of an Al film or an Al alloy film is formed. Thereby, an Al interconnection film 8 of the DD structure is constituted. In the figure, reference numeral 9 denotes an insulating film.

The Al interconnection film 8 having the Nb film 6

deposited by use of the present sputtering target, as mentioned above, satisfies the resistivity as low as 4μΩcm or less. Accordingly, performance characteristic or reliability of the semiconductor devices such as DRAMs of for instance 256Mbit or 1Gbit or more can be improved. Further, the giant dust being scarcely mixed, the high density interconnection can be realized with high reliability and reproducibility.

Next, concrete embodiments of the present invention and evaluation results thereof are described.

Embodiment 1

First, Nb_2O_5 containing concentrate of which Ta content is 3000ppm or less is chemically processed to be a high purity oxide, the oxide being reduced by making use of thermite reduction method due to Al to obtain crude metal Nb. Several pieces of such crude metal Nb are prepared. These are EB melted appropriately between one to multiple times to prepare 6 kinds of Nb ingots (diameter of 230mm) different in the Ta content.

Each of these Nb ingots is drawn and forged to be a diameter of 130mm, being annealed at a temperature of 1400°C, thereafter being forged again to be a diameter of 230 to 240mm, and further being rolled by use of cross-rolling to be a disk of a diameter of 320 to 330mm. To these disc-like Nb plates, heat treatment is applied under the conditions of 1100°C × 120min to recrystallize.

The respective Nb plates after the aforementioned heat treatment are cut to be Nb plates for bonding, these and Al

alloy plates for backing plate prepared in advance being hot pressed under the conditions of a temperature of 400 to 600°C and a pressure of 250kg/cm^2 to be bonded bodies that are target raw material. Each of thus obtained bonded bodies is machined to be a diameter of 320mm × a thickness of 10mm, thereby an intended Nb sputtering target being obtained.

The Ta contents and dispersions thereof are measured of thus obtained 6 kinds of Nb targets based on the aforementioned method. The Ta content is analyzed by use of usually used ICP-AES (Inductively Coupled Plasma Atomic Emission Spectrometry apparatus: Seiko Instrument Industries' product SPS1200A (Commercial Name)). The Ta contents and dispersion thereof in the Nb targets are shown in Table 1.

Next, with each of the aforementioned 6 kinds Nb sputtering targets, under the conditions of sputtering method of liner sputtering, a back pressure of 1×10^{-5} (Pa), a DC output of 15 (kW) and a sputtering time of 1 (min), on a Si wafer (8 inches) with previously formed interconnection trench a Nb film is formed. Thereby, a liner film of a thickness of 0.5μm is formed including the inside of the interconnection trench. Thereafter, with an Al-0.5% by weight of Cu target, the sputtering is implemented under the conditions identical with the aforementioned conditions to form an Al thin film of a thickness of approximately 1μm. After the Al thin film is filled in the interconnection trench due to the reflow treatment, an excess of the Al film is removed due to the CMP to form an interconnection. Each resistivity of these interconnections is measured. The

results are shown in Table 1.

Table 1

Target No.	Ta Content (ppm)	Dispersion of Ta Content (%)	Resistivity of Interconnection ($\mu\Omega$cm)
No.1	550	11	3.1
No.2	1550	27	3.5
No.3	1830	40	3.9
No.4	2540	5	3.8
No.5	3300	17	10.5
No.6	8220	58	15.8

As obvious from Table 1, it is found that interconnection films each comprising a Nb film formed by use of each of the present Nb targets of specimen 1 to specimen 4 are one half or less in resistivity compared with that of the other interconnection films. By employing the interconnection film having such Nb liner film, the interconnection can be reduced in the resistivity, and further product yield can be largely improved.

Embodiment 2

Crude metal Nb prepared similarly with Embodiment 1 is EB melted three times to prepare an ingot. The Nb ingot undergoes plastic working under the conditions identical with Embodiment 1, thereafter by varying the heat treatment condition 6 kinds of Nb materials are prepared. The heat treatment temperatures are 300°C, 600°C, 800°C, 1100°C and 1300°C, and treatment time periods are 60min for all heat treatments. In addition, Nb material that is not heat-treated is prepared.

With such 6 kinds of Nb materials, similarly with

Embodiment 1 Nb sputtering targets are prepared, respectively. The Ta content is analyzed due to an IPC-AES similarly with Embodiment 1. The Ta content is 1830ppm, the dispersion thereof being 20%.

Next, with each of the aforementioned 6 kinds Nb sputtering targets, under the conditions of sputtering method of liner sputtering, a back pressure of 1×10^{-5} (Pa), a DC output of 15 (kW) and a sputtering time period of 1 (min), on a Si wafer (8 inches) with previously formed interconnection trench a Nb film is formed. Thereby, a liner film of a thickness of 0.5μm is formed including the inside of the interconnection trench. Thereafter, with an Al-0.5% by weight of Cu target, the sputtering is implemented under the conditions identical with the aforementioned conditions to form an Al thin film of a thickness of approximately 1μm. After the Al thin film is filled in the interconnection trench due to the reflow treatment, an excess of the Al film is removed due to the CMP to form an interconnection. Each resistivity of these interconnections is measured. The results are shown in Table 2.

Table 2

Target No.	Heat Treatment Temperature (°C)	Resistivity of Interconnection ($\mu\Omega$cm)
No.1	Without heat treatment	3.8
No.2	300	3.7
No.3	600	3.5
No.4	800	3.2
No.5	1100	3.1
No.6	1300	3.1

As obvious from Table 2, interconnection films each comprising a Nb film formed by use of the present Nb sputtering target show excellent resistivity. Accordingly, by use of the interconnection film having such Nb liner film, the interconnection can be made low in the resistivity, and the product yield can be largely improved.

Embodiment 3

First, crude metals of Nb are prepared by varying oxygen contents. These are EB melted appropriately between one to multiple times to prepare 6 kinds of Nb ingots (diameter of 230mm) of different oxygen contents.

Each of these Nb ingots is drawn and forged to be a diameter of 130mm, being annealed at a temperature of 1400°C, thereafter being forged again to be a diameter of 230 to 240mm, and further being rolled by use of cross-rolling to be a disk of a diameter of 320 to 330mm. To these disc-like Nb plates, heat treatment is applied under the conditions of 1100°C × 120min to recrystallize.

The respective Nb plates after the aforementioned heat treatment are cut to be Nb plates for bonding, these and Al alloy plates for backing plate prepared in advance being hot pressed under the conditions of a temperature of 400 to 600°C and a pressure of 250kg/cm^2 to be bonded bodies that are target raw material. Each of thus obtained bonded bodies is machined to be a diameter of 320mm × a thickness of 10mm, thereby an intended Nb sputtering target being obtained.

The oxygen contents and dispersions thereof are

measured of thus obtained 6 kinds of Nb targets based on the aforementioned method. The oxygen content is analyzed by use of usually used inert gas melting infrared absorption (LECO' product TC-436 (Commercial Name)). The oxygen contents and dispersions thereof are shown in Table 3.

Next, with each of the aforementioned 6 kinds Nb sputtering targets, under the conditions of sputtering method of liner sputtering, a back pressure of 1 $\times$ 10^{-5} (Pa), a DC output of 10 (kW) and a sputtering time of 3 (min), on a Si wafer (8 inches) with previously formed interconnection trench a Nb film is formed. Thereby, a liner film of a thickness of 20μm is formed including the inside of the interconnection trench. Thereafter, with an Al-0.5% by weight of Cu target, the sputtering is implemented under the conditions identical with the aforementioned conditions to form an Al thin film of a thickness of approximately 1μm. After the Al thin film is filled in the interconnection trench due to the reflow treatment, an excess of the Al film is removed due to the CMP to form an interconnection. Each resistivity of these interconnections is measured. The results are shown in Table 3.

Table 3

Target No.	Oxygen Content (ppm)	Dispersion of Oxygen Content (%)	Resistivity of Interconnection ($\mu\Omega$cm)
No.1	10	±82	4.2
No.2	10	±40	3.0
No.3	50	±23	3.1
No.4	60	±64	3.1
No.5	100	±27	3.2
No.6	110	±68	3.4
No.7	140	±38	3.5
No.8	320	±31	4.1
No.9	630	±22	4.4
No.10	820	±20	4.7

As obvious from Table 3, it is found that interconnection films each comprising a Nb film deposited by use of each of the present Nb targets of specimen 2 to specimen 7 are lower in the resistivity compared with that of the other interconnection films. By employing the interconnection film having such Nb liner film, the interconnection can be reduced in the resistivity, and further product yield can be largely improved.

Embodiment 4

Under the conditions identical with that of the target of specimen 3 of Embodiment 3, a Nb ingot is prepared. The Nb ingot undergoes plastic working under the conditions identical with that of Embodiment 3, by varying the heat treatment conditions, 6 kinds of Nb materials are prepared. The heat treatment temperatures are 300°C, 600°C, 800°C, 1100°C and 1300°C, and treatment time periods are 60min for all heat treatments. Nb material that is not heat-treated is

prepared.

With each of 6 kinds of Nb materials, similarly with Embodiment 1 Nb targets are prepared respectively. The oxygen content, as the result of measurement in the identical way with Embodiment 3, shows a value of approximately identical with specimen No. 3 in Embodiment 3.

Next, with each of the aforementioned 6 kinds Nb sputtering targets, under the conditions of sputtering method of liner sputtering, a back pressure of 1×10^{-5} (Pa), a DC output of 10 (kW), a sputtering time of 3 (min), on a Si wafer (8 inches) with previously formed interconnection trench a Nb film is formed. Thereby, a liner film of a thickness of 20μm including the inside of the interconnection trench is formed. Thereafter, with an Al-0.5% by weight of Cu target, the sputtering is implemented under the conditions identical with the aforementioned conditions to form an Al thin film of a thickness of approximately 1μm. After the Al thin film is filled in the interconnection trench due to the reflow treatment, an excess of the Al film is removed due to the CMP to form an interconnection. Each resistivity of these interconnections is measured. The results are shown in Table 4.

Table 4

Target No.	Heat Treatment Temperature (°C)	Resistivity of Interconnection ($\mu\Omega$cm)
No.1	Without heat treatment	4.9
No.2	300	4.3
No.3	600	3.8
No.4	800	3.1
No.5	1100	2.9
No.6	1300	3.0

As obvious from Table 4, interconnection films each comprising a Nb film formed by use of each of the present Nb targets (specimen 3 to specimen 6) are excellent in resistivity. Accordingly, by employing the interconnection film having such Nb liner film, the interconnection can be reduced in the resistivity, and further product yield can be largely improved.

Embodiment 5

First, crude metal of Nb prepared identically with Embodiment 1 is EB melted to prepare an ingot of Nb of a diameter of 230mm. To such the Nb ingot, forging and rolling similar with that of Embodiment 1 are implemented. However, by varying working conditions at each stage, with the respective working rates shown in Table 5, the plastic working is implemented. The working rate is calculated by use of the formula of {100-(thickness after plastic working/height of ingot) × 100}.

To thus obtained disc-like Nb plates, heat treatment is applied under the conditions of 1100°C × 120min to recrystallize. The respective Nb plates are cut to be Nb plates for bonding, these and Al alloy plates for backing

plate prepared in advance being hot pressed under the conditions of a temperature of 400 to 600°C and a pressure of 250kg/cm^2 to be bonded bodies that are target raw material. Each of thus obtained bonded bodies is machined to be a diameter of 320mm × a thickness of 10mm, thereby an intended Nb sputtering target being obtained.

Next, with each of the aforementioned Nb sputtering targets, under the conditions of sputtering method of DC sputtering, a back pressure of 1 × 10^{-5} (Pa), a DC output of 15 (kW) and a sputtering time of 1 (min), on a Si wafer (8 inches) a Nb film of a thickness of 0.5μm is formed. 500 plates of Si substrates are deposited, respectively.

For the respective Nb films, a range of grain diameters relative to an average grain diameter of Nb grains, ratios of grain sizes of adjacent grains and dispersion thereof are measured. Further, number of the giant dust of a size of 1 μm or more in each Nb film is measured. These results are shown in Table 5.

Table 5

Target No.	Working Rate (%)	Average Grain Diameter (μm)	Range of Grain Diameter to Average Grain Diameter (%)	Grain Size Ratio of Adjacent Grains		Average Number of Dust (1μm or more) (Pieces /Plate)
				Ratio of Grain Size	Dispersion in a Target (%)	
1	95	30	0.7	0.6	2	0
2	87	70	4.5	1.2	5	0
3	55	100	7.8	5.8	12	0
4	25	190	0.05	5	35	0.8
5	33	280	15.8	17	45	0.6
6	14	350	23.2	58	67	1.2

As obvious from Table 5, it is found that in the Nb films deposited with the present Nb sputtering targets (specimen No. 1 to 3), no giant dust exists, by contrast, in the Nb sputtering targets (specimen No. 4 to 6) prepared as the comparative examples of the present invention, the giant dust exists in the Nb films deposited therewith. Accordingly, by employing the Nb films of the present invention like this, the yield of the interconnection films and various devices therewith can be largely improved.

Embodiment 6

Similarly with Embodiment 5, to Nb plates thereto forging and rolling are applied with the working rate of 85%, no heat treatment, or 300°C, 600°C, 800°C, 1100°C and 1300°C heat treatments for 60min each are applied to prepare 6 kinds of Nb materials. With these 6 kinds of Nb materials, similarly with Embodiment 1, the Nb sputtering targets are prepared, respectively.

Next, with each of the aforementioned Nb sputtering targets, under the conditions of sputtering method of DC sputtering, a back pressure of 1×10^{-5} (Pa), a DC output of 15 (kW) and a sputtering time of 1 (min), on a Si wafer (8 inches) a Nb film of a thickness of 0.5μm is formed. 500 plates of Si substrates are deposited, respectively.

For the respective Nb films, a range of grain diameters relative to an average grain diameter of Nb grains, ratios of grain sizes of adjacent grains and dispersion thereof are measured. Further, number of giant dust of a size of 1 μm or more in each Nb film is measured. These results are shown

in Table 6.

Table 6

Target No.	Heat Treatment Temperature (°C)	Average Grain Diameter (μm)	Range of Grain Diameter to Average Grain Diameter (%)	Grain Size Ratio of Adjacent Grains		Average Number of Dust (1μm or more) (Pieces/ Plate)
				Ratio of Grain Sizes	Dispersion in a Target (%)	
1	Without Treatment	–	–	–	–	3.5
2	300	–	–	–	–	1.2
3	600	–	–	–	–	0.1
4	800	10	0.5	7.8	1.2	0
5	1100	30	1.3	1.1	4.4	0
6	1300	70	4.7	0.7	12	0

As obvious from Table 6, in the Nb films deposited with the present Nb sputtering targets (specimen No. 4 to 6), there is found no giant dust. By contrast, in the Nb sputtering targets (specimen No. 1 to 3) prepared as the comparative examples of the present invention, there is found the giant dust in the Nb films deposited therewith. Accordingly, by employing the Nb films of the present invention like this, the yield of the interconnection films and various devices therewith can be largely improved.

Industrial Applicability

A Nb sputtering target of the present invention enables to obtain an interconnection film of low resistivity that has not been previously achieved. Alternatively, the present Nb sputtering target enables with reproducibility to suppress giant dust from occurring. Accordingly, according to an

interconnection film deposited with such sputtering target, an interconnection of low resistivity can be achieved and further reliability or yield can be largely improved.

While in the foregoing specification this invention has been described in relation to certain preferred embodiments thereof, and many details have been set forth for purpose of illustration, it will be apparent to those skilled in the art that the invention is susceptible to additional embodiments and that certain of the details described herein can be varied considerably without departing from the basic principles of the invention.

Claims

(Amended on June 20, 2000 under PCT Article 34)

1. (Amended) A sputtering target consisting essentially of high purity Nb of which Ta content is 3000ppm or less, wherein dispersion of the Ta contents over the whole target is within ±30%.

2. (Deleted).

3. The sputtering target as set forth in claim 1: wherein the Ta content is 1000ppm or less.

4. The sputtering target as set forth in claim 1: wherein oxygen content in the target is 200ppm or less.

5. The sputtering target as set forth in claim 1: wherein an average grain diameter of the Nb is 100μm or less.

6. The sputtering target as set forth in claim 1: wherein each grain of the Nb has a grain diameter in the range of 0.1 to 10 times an average grain diameter, and each of ratios of grain sizes of adjacent grains is in the range of 0.1 to 10.

7. The sputtering target as set forth in claim 1: wherein the sputtering target is bonded with a backing plate.

8. The sputtering target as set forth in claim 7: wherein the sputtering target and the backing plate are diffusion bonded.

9. The sputtering target as set forth in claim 1: wherein the sputtering target is applied in forming a

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Nb film as liner material to an Al film or an Al alloy film.

10. A sputtering target consisting essentially of high purity Nb:

wherein each grain of the Nb has a grain diameter in the range of 0.1 to 10 times an average grain diameter, and each of ratios of grain sizes of adjacent grains is in the range of 0.1 to 10.

11. The sputtering target as set forth in claim 10:

wherein dispersion of the ratios of grain sizes of the adjacent grains all over the target is within ±30%.

12. The sputtering target as set forth in claim 10:

wherein each of the ratios of the grain sizes of the adjacent grains is in the range of 0.5 to 5.

13. The sputtering target as set forth in claim 10:

wherein an average grain diameter of the Nb is 100μm or less.

14. The sputtering target as set forth in claim 10:

wherein oxygen content in the target is 200ppm or less.

15. The sputtering target as set forth in claim 10:

wherein the sputtering target is bonded with a backing plate.

16. The sputtering target as set forth in claim 15:

wherein the sputtering target and the backing plate are diffusion bonded.

17. The sputtering target as set forth in claim 11:

wherein the sputtering target is used in forming a Nb film as liner material to an Al film or an Al alloy film.

18. (Amended) A sputtering target consisting

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essentially of high purity Nb of which oxygen content is 200ppm or less, wherein dispersion of the oxygen contents over the whole target is within ±80%.

19. (Deleted).

20. The sputtering target as set forth in claim 18: wherein the oxygen content is 100ppm or less.

21. The sputtering target as set forth in claim 18: wherein the sputtering target is bonded with a backing plate.

22. The sputtering target as set forth in claim 21: wherein the sputtering target and the backing plate are diffusion bonded.

23. The sputtering target as set forth in claim 18: wherein the sputtering target is used for forming a Nb film as liner material to an Al film or an Al alloy film.

Abstract

A sputtering target consists of high purity Nb of which Ta content is 3000ppm or less and oxygen content is 200ppm or less. Dispersion of the Ta content in all the sputtering target is within ±30% as a whole target. Dispersion of the oxygen content is within ±80% as a whole target. According to such sputtering target, an interconnection film of low resistivity can be realized. In addition, each grain of Nb in the sputtering target has a grain diameter in the range of 0.1 to 10 times an average grain diameter and ratios of grain sizes of adjacent grains are in the range of 0.1 to 10. According to such sputtering target, giant dust can be largely suppressed from occurring. The sputtering target is suitable for forming a Nb film as liner material of an Al interconnection.

Title: SPUTTERING TARGET
Inventor(s): Koichi WATANABE et al.
DOCKET NO.: 017447/0170

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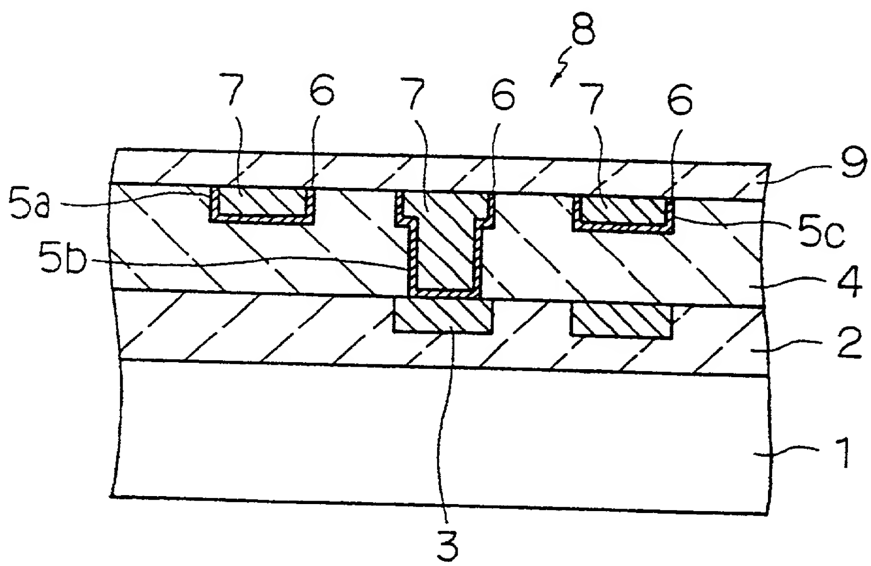

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Japanese Language Declaration

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____________________	SPUTTERING TARGET
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(Slight Modification was made at priority claiming portion.)

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I hereby claim foreign priority under Title 35, United States Code, Section 119(a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application(s)
外国での先行出願

(Number) (番号)	(Country) (国名)	(Day/Month/Year Filed) (出願年月日)	Priority Claimed 優先権主張 Yes はい	No いいえ
P10-182689	Japan	29/June/1998	☒	☐
P10-204001	Japan	17/July/1998	☒	☐
P10-212829	Japan	28/July/1998	☒	☐

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(Application No.) (出願番号)	(Filing Date) (出願日)	(Application No.) (出願番号)	(Filing Date) (出願日)

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第二共同発明者の署名 日付	Second inventor's signature Date Yasuo Kohsaka 24-Nov.-2000
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（第三以降の共同発明者についても同様に記載し、署名をすること）

(Supply similar information and signature for third and subsequesnt joint inventors.)

Japanese Language Declaration

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